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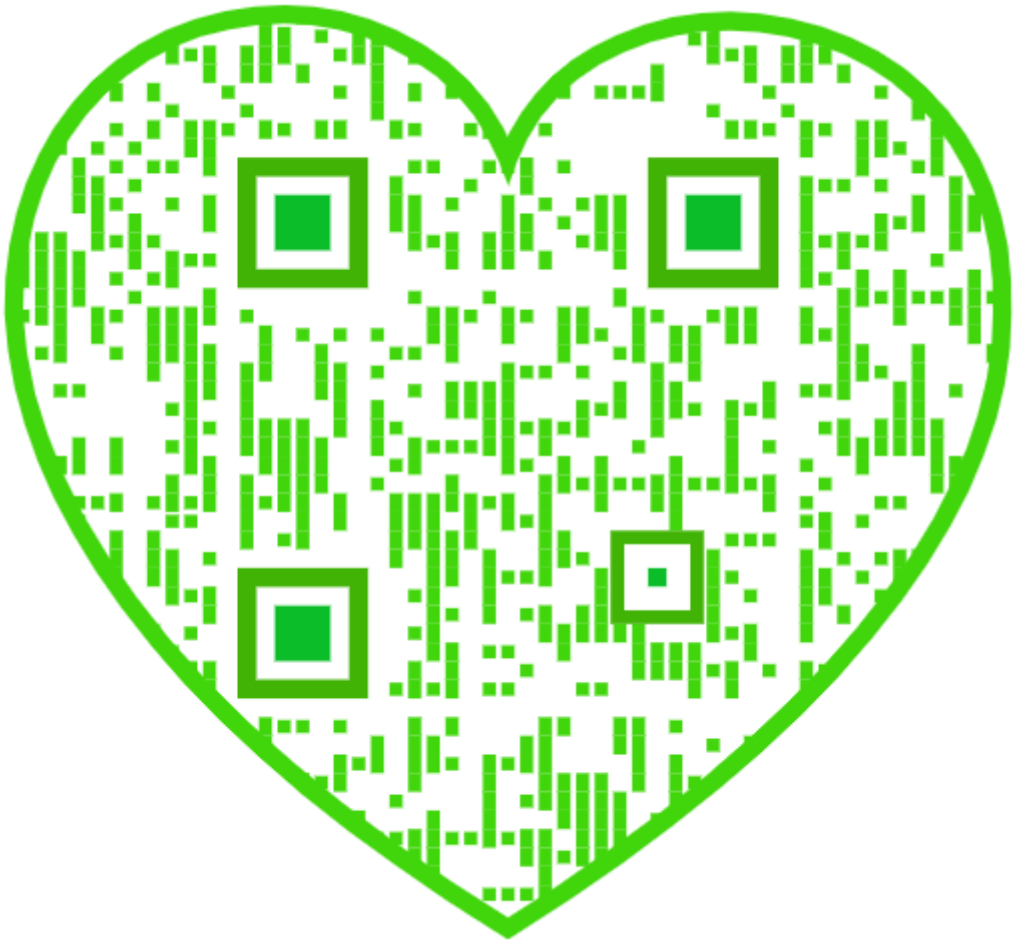

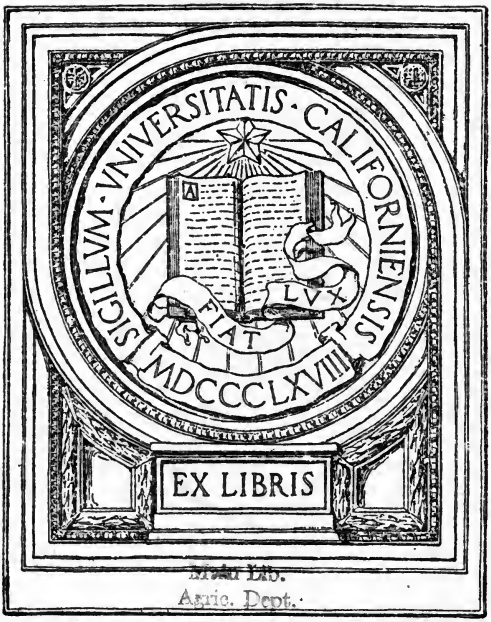

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United States Department of Agriculture,

BUREAU OF CHEMISTRY—Circular No. 33.

H. W. WILEY, Chief of Bureau.

REPORT ON METHODS OF BEER ANALYSIS.

BY H. E. BARNARD,

Chemist Indiana State Board of Health, and Associate Referee on Food Adulteration, Association of Official Agricultural Chemists.

INTRODUCTION.

The methods for the analysis of beers having been studied for several years by the Association of Official Agricultural Chemists, the work has culminated in the following exhaustive report submitted by the referee at the convention held in 1906, and the methods reported are now before the association for adoption as official. For this reason, as well as because of the length of the report and the fact that while important it is of interest to a comparatively limited number of chemists, the methods are here published in circular form.

Respectfully,

H. W. WILEY,
Chief, Bureau of Chemistry.
Secretary, Association of Official Agricultural Chemists.

Approved:

JAMES WILSON,
Secretary of Agriculture.

WASHINGTON, D. C., *January 16, 1907.*

21185—No. 33—07

ORGANIZATION OF THE COOPERATIVE WORK, 1906.

At the meeting of the Association of Official Agricultural Chemists in 1904, tentative methods for beer analysis were reported and published in the Proceedings for that year. Since then similar analytical methods have been submitted to all of the brewing schools of the country and to the committee of scientists of the United States Brewers' Association. The correspondence which followed developed many interesting and valuable points and brought out several new analytical methods that seemed worthy of study. In May, 1906, there was sent to eight members of the association and five brewing institutes and laboratories, who had signified their willingness to cooperate in beer work, the following letter, accompanying samples as described and giving methods for analysis to be used in the investigations:

DEAR SIR: In response to your offer to do cooperative work on beer analysis, I am inclosing herewith directions for the examination of samples of beer which are being shipped you by express. The methods as suggested explain themselves, I believe.

My desire is to obtain full reports on the two samples for the purpose of comparing results obtained by the different methods, omitting, if you wish, the glycerol and carbon dioxid results. The samples are numbered 1 and 2. One of the samples is preserved with sodium sulphite and the other with sodium fluorid. The beers have been pasteurized and will keep for some weeks in a normal condition if in a cool place.

I shall be glad to receive your reports on the samples, together with such comments as you may make on the various methods as to their accuracy, practicability, etc., at an early date.

Thanking you for your willingness to aid me in this work, I am,

Very truly yours,

H. E. BARNARD,
Associate Referee on Beer.

PROVISIONAL METHODS FOR BEER ANALYSIS.

[Submitted to collaborators for trial, 1906.]

At present there is a decided lack of uniformity in the methods employed in the analysis of fermented beverages. Food chemists rely for the most part on the provisional methods published in Bulletin No. 65 of the Bureau of Chemistry, Department of Agriculture.[a] Chemists in the brewing industry use certain other methods, and there is a lack of agreement as to the most suitable and accurate method of analysis. In order to decide upon standard methods as fairly and accurately as possible it will be necessary to give each of the many different methods employed for the determination of the various factors a thorough trial. It is therefore, recommended that in making analyses of the samples the directions be followed as closely as possible and results expressed in the form indicated.

PREPARATION OF SAMPLE.

Bottled beers are always under pressure because of the carbon dioxid present, either as the result of natural fermentation or artificial carbonating. To remove all undissolved carbon dioxid (A) pour contents of bottle into a large flask and shake vigorously to hasten the escape of gas; (B) drive off gas by pouring sample from one tall beaker to another until on standing no gas bubbles are seen to rise. In preparing the sample see that the temperature is not lower than 15° C.

Object: To determine, first, whether method (A) secures as complete a separation of carbon dioxid as (B) and, second, to see if any loss of alcohol is observed when samples are exposed to open air, as in method (B).

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[a] Provisional methods for the analysis of foods, p. 92.

Specific Gravity.

Determine specific gravity of sample after removal of carbon dioxid by means of the pycnometer, by a small, accurately graduated hydrometer, by a Westphal balance, or by a Westphal plummet on the analytical balance. Care should be taken when using the hydrometer that the liquid is quickly raised to room temperature without loss by evaporation, after filling and before weighing, to prevent condensation of moisture on the outside of the flask.

A. Determine specific gravity at 15° C.

B. Determine specific gravity at 20° C.

Calculate apparent extract or balling of beer according to Schultz and Ostermann's table, page 127, Bulletin 65; Ellion's table, page 132, Bulletin 65, and Balling's extract table if available.

Object: To determine errors introduced by use of different tables.

Alcohol.

A. Measure 100 cc of the liquid at 20° C. into a round-bottomed distillation flask, add 50 cc of distilled water, and if the beer is markedly acid, 0.1 or 0.2 gram of precipitated calcium carbonate, connect with condenser and distil off 90 cc into an accurately graduated 100 cc stopped flask, care being taken to prevent loss of alcohol during the distillation. This is best accomplished by inserting a two-holed rubber stopper in the mouth of the flask, through one hole of which passes an adapter which is tightly fitted to the end of the condenser and through the other a small funnel which is filled with glass beads kept moist by sprinkling water. Wash down the beads several times during the distillation with a small quantity of water, thus saving any alcohol which may have collected on the moist surface. Bring the contents of the receiver to 20° C., make up to the mark with distilled water, determine the specific gravity as directed under "Specific gravity," and obtain the corresponding percentage of alcohol by volume or grams per 100 cc from Squibb's alcohol table (p. 121, Bull. 65), or Baumhauer's tables, if at hand. Calculate the results to per cent by weight by dividing the results expressed as grams per 100 cc by the specific gravity of the original sample.

B. When the original extract of the wort is known the alcohol content of the beer can be calculated by the following formula: $A = 0.42\ (B_1 - B_2)$ when A = alcohol, B_1 the original extract of the wort, and B_2 the extract of the beer.

C. Dealcoholize 100 cc of the beer in a porcelain dish on the water bath, make up to 100 cc and take the specific gravity, then $A = \frac{B_1}{B_2}$ when A = specific gravity of alcohol, B_1 the specific gravity of beer, and B_2 the specific gravity of the dealcoholized beer.

D. Calculate the alcohol content from the reading of the Zeiss immersion refractometer on the distillate at a definite temperature.

E. Calculate from the formula $A = (B_1 - B_2) + 15$, when A = reading on immersion refractometer, B_1 reading of beer, and B_2 reading of dealcoholized beer, and determine value for A from alcohol tables accompanying the instrument.

Object: To determine most accurate method commensurate with rapidity of determination.

Extract.

A. Evaporate 25 cc of beer in a tared platinum dish to constant weight in a water oven at 80° C.

B. Calculate according to formula, $sp = g + (1 - a)$ in which sp is the specific gravity of the dealcoholized beer, g the specific gravity of the beer, and a the specific gravity of the distillate obtained in the determination of alcohol, and determine value of sp from extract in beer wort tables, pages 127 and 132, Bulletin 65.

C. Dilute the residue from the distillation of alcohol to original volume, take the specific gravity, and determine value from tables as in paragraph B.

D. Take immersion refractometer reading and calculate extract from tables in grams per 100 cc.

Object: To determine whether or not method (A) gives incorrect results because of dehydration of maltose.

Extract and Specific Gravity of Wort.

Various methods are employed to obtain these figures, all of which are based on the amount of alcohol produced by the fermentation of a given quantity of sugar.

Calculate from the following formulas:

A. O=2 A+E, when O=original extract of wort, A=alcohol by weight, and E=extract of dealcoholized beer.

B. O=(A×1.92)± E. Same as formula A, except that factor for converting alcohol to sugar is theoretically more accurate.

C. $O=E+\frac{E-A}{1.232-1}$ when E=real extract and A=apparent extract.

D. $O=100\times\frac{(2.0665\times A)+E}{(1.0665\times A)+100}$ when A=alcohol and E=extract.

E. G=*sp*+*si*, where G is the specific gravity of the original wort, *sp* the specifi-gravity of the dealcoholized beer, and *si* the amount of alcohol destroyed by fermentation, as obtained from table in Allen's Commercial Organic Analysis, volume 1, page 135.

From extract calculated as in A, B, C, and D, compute from Schultz and Ostermann's, Ellion's, and Balling's tables the specific gravity of the wort.

Degree of Fermentation.

Calculate from the formula $D=\frac{200\ A}{B}$ in which D is the degree of fermentation, A the percentage of alcohol by weight, and B the original extract.

Total Acids.

A. Heat 20 cc of the sample to incipient boiling to liberate carbon dioxid and titrate with decinormal sodium hydroxid, using neutral litmus paper as indicator. Each cubic centimeter of decinormal alkali employed is equivalent to 0.009 gram of lactic acid. The number of cubic centimeters of decinormal alkali employed in titrating 20 cc of the beer is multiplied by 0.045 for the acidity expressed as grams of lactic acid per 100 cc.

B. Calculate as cubic centimeters of decinormal sodium hydroxid required to neutralize the acidity of 100 cc of the sample.

Volatile Acids.

A. The volatile acid as acetic acid is determined by titrating 20 cc of the alcohol distillate with decinormal sodium hydroxid solution, using phenolphthalein as an indicator. The number of cubic centimeters of decinormal alkali employed multiplied by 0.030 gives the acidity expressed as grams of acetic acid per 100 cc.

B. Calculate as cubic centimeters of decinormal sodium hydroxid required to neutralize the acidity of 100 cc of the sample.

Reducing Sugars.

Twenty-five cubic centimeters of the beer free from carbon dioxid are diluted with water to 100 cc. The reducing sugar is determined in 25 cc of this solution, as directed on page 49, Bulletin 65, the solution being boiled 4 minutes instead of 2 minutes. Express the results in terms of maltose equivalent to the copper reduced, according to Table IX, page 144, Bulletin 65.

Dextrin.

A. Employ Sachsse's method for the hydrolization of starch and determine dextrose according to Allihn, as follows:

Fifty cubic centimeters of beer and 15 cc of hydrochloric acid, specific gravity 1.125, are diluted to 200 cc, attached to a reflux condenser and kept in a boiling water bath for 2 hours. Neutralize with caustic soda and dilute to 250 cc (or 300 cc in a beer with high extract). Add 25 cc of the solution to the boiling Fehling solution and boil 2 minutes. Multiply the oxid of copper found by 0.8 to obtain the corresponding amount of copper and refer to Allihn's dextrose table (Table VIII, p. 143, Bull. 65). The amount of dextrose thus found multiplied by 20 (or 24 if diluted to 300 cc) and divided by the specific gravity equals the dextrose in the original beer. From this figure subtract 95 per cent of the amount of maltose in the original beer and multiply the remainder by 0.9, the result being the percentage of dextrin in the original beer.

B. Dextrin may be determined by the following differential method based on the difference between its optical activity and that of maltose:

$D=\frac{A-(M\times 8.1)}{11.6}$ when D=dextrin in grams per 100 cc; A=total rotation in degrees Ventzke in 200 mm tube, and M= percentage of maltose as determined gravimetrically.

DIRECT POLARIZATION.

Read the polarization of the original sample in degrees Ventzke in a 200 mm tube. If the beer is turbid clarify by shaking with alumina cream.

INVERT POLARIZATION.

To 10 cc of the beer in a small flask add 1 cc of concentrated hydrochloric acid, invert by slowly heating to 68° C., cool, polarize in a 200 mm tube and increase the reading one-tenth to allow for dilution.

GLYCEROL.

Proceed as directed on page 82, Bulletin 65. The milk of lime is added during evaporation after the carbon dioxid has been expelled. It is advisable that the filtrate, after being evaporated to a sirupy consistency, be treated again with 5 cc of absolute alcohol and two portions of 7.5 cc each of absolute ether. If clear, continue as directed; if not clear, it is necessary to treat as above.

ASH.

Evaporate 25 cc of the sample to dryness, ignite at low redness until thoroughly charred, place in a muffle, and continue the ignition until ash is white. Leaching is rarely necessary.

PHOSPHORIC ACID.

Measure out 50 cc of the beer, free from carbon dioxid, into a small beaker. Add 5 cc of an acid solution of sodium acetate and heat to boiling. Run in from a burette, standard uranium acetate solution 0.5 cc at a time, testing each time until a drop of the beer, when placed on a white plate, colors a drop of potassium ferrocyanid slightly brown. The number of cubic centimeters of the uranium acetate solution necessary, multiplied by 0.01, gives the grams per 100 cc of phosphoric acid in the beer. If the beer is very dark, employ the official method, gravimetric or volumetric, using the residue obtained in the determination of the ash.

PROTEIN.

Evaporate 25 cc of the original beer, to which has been added a small amount of tannin to prevent frothing, and proceed according to the Kjeldahl or Gunning method for the determination of nitrogen, and multiply the result by 6.25.

CARBON DIOXID.

Proceed as directed in Bulletin 65, page 95.

PRESERVATIVES.

Add 5 cc of dilute sulphuric acid to the remaining residue after distillation, and shake out with an equal volume of a mixture of ether and benzene. Pour off the supernatent liquid and test for salicylic acid, benzoic acid, and saccharin in the usual manner. It is well to reserve 2 or 3 cc of the liquid in a test tube for the salicylic acid test before evaporating the remainder to dryness in a small porcelain evaporating disk.

Determine sulphites and sulphurous acid as determined under wines, page 90, Bulletin 65, employing the method of direct determination on 50 cc of the sample.

Determine flourids by the method of Blarez as follows: Thoroughly mix the sample and heat 150 cc of the sample to boiling. Add to the boiling liquor 5 cc of a 10 per cent solution of barium acetate. Collect the precipitate in a compact mass, using to advantage a centrifuge, wash upon a small filter, and dry in the oven. Transfer to a platinum crucible, first breaking up the dry precipitate and then adding the filter ash to the crucible.

Prepare a glass plate (preferably of the thin variety commonly used for lantern-slide covers) as follows: First, thoroughly clean and polish, and coat on one side by carefully dipping while hot in a mixture of equal parts of Carnaüba wax and paraffin. Near the middle of the plate make a small cross or other distinctive mark through the wax with a sharp instrument, such as a pointed piece of wood or ivory, which will remove the wax and expose the glass without scratching the latter. Add a few drops of concentrated sulphuric acid to the residue in the crucible and cover the crucible with the waxed plate, having the mark nearly over the center and making sure that the crucible is firmly embedded in the wax. Place in close contact with the top or unwaxed surface of the plate a cooling device consisting of a glass tube covered tightly with a thin sheet of pure rubber. A constant stream of cold water is passed continually through the tube. The whole arrangement is lowered so that the crucible rests upon a heated electric stove, where it remains for an hour.

Remove the glass plate and indicate the location of the distinguishing mark on the unwaxed surface of the plate by means of gummed strips of paper, and melt off the wax by heat or a jet of steam and thoroughly clean the glass with a soft cloth. A distinct etching will be apparent on the glass where it was exposed if a fluorid be present.

ANALYTICAL RESULTS.

The accompanying tables give the results obtained by the different analysts on the two samples sent out. The various factors determined are reported in full.

TABLE 1.—*Analytical results obtained by cooperating chemists on referee's sample of beer No. 1.*

[Results calculated to grams per 100 cc except where otherwise stated.]

Determinations.	W. M. Cross.	Francis Wyatt.	Max Henius.	Frank Kremer.	B. Breitenfeld.
1. Specific gravity at 15° C	1.0105	1.0111	1.0110	1.0113	1.0109
2. 20° C	1.0095	1.0109	1.0107	1.0112	1.0108
3. Alcohol A: by distillation (per cent by weight)	3.75	3.88	3.91	3.74	3.93
4. C: $A=B_1 \div B_2$ (per cent by weight)	3.92	3.70	.....	3.72	3.83
5. D: = by the immersion refractometer.	3.87	3.87	3.83	.....	3.83
6. E: by $A=(B_1-B_2)+15$	3.52	.....	.....	.....	.....
7. Extract A: by direct weighing	4.69	4.60	4.38	4.59	4.53
8. B: Sp.=g+(1−a)	4.55	4.68	4.71	4.66	4.49
9. C: from dealcoholized beer	4.66	4.72	4.64	4.69	4.48
10. D: by immersion refractometer	4.75	.....	.....	.....	4.50
11. Extract of wort A: O=2A+E	12.19	12.46	12.48	11.93	12.34
12. B: O=(A×1.92)+E	11.89	12.15	12.16	11.63	12.02
13. C: O=E+[(E−A)÷(1.232−1)]	.....	12.73	12.43	11.78	.....
14. D: O=100[(2.0665×A)+E]÷[(1.0665×A)+100]	.....	12.31	12.23	11.53	.....
15. E: G=sp+si	11.35	11.80	12.05	.....	.....
16. Specific gravity, Shultz & Ostermann's tables	1.0475	1.0480	1.0481	1.0471	1.0454
17. Ellion's table	1.0504	1.0496	1.0460	1.0500	1.0480
18. Balling's table	1.0496	1.0486	1.0488	1.0485	1.0489
19. Degree of fermentation: (200A)÷B	62.5	62.4	62.7	62.3	65.4
20. Total acids A: gram lactic acid per 100 cc	.124	.099	.058	.081	.11
21. B: cc $\frac{n}{2}$ NaOH to 100 cc	13.8	11.0	6.5	9.0	12.5
22. Volatile acids A: gram acetic acid per 100 cc	.004	.009	.0046	.003	.006
23. B: cc $\frac{n}{10}$ NaOH to 100 cc	.7	1.5	.8	.5	1.0
24. Reducing sugars	1.567	1.55	1.53	1.40	1.51
25. Dextrin A: by Allihn's method	2.3	.....	.....	.....	2.19
26. B: =(A−(M×8.1))÷(11.6)	1.38	.....	.....	.....	1.83
27 Direct polarization: °V	28.5	.....	.....	.....	32.4
28. Invert polarization: °V	23.2	.....	.....	.....	31.6
29. Glycerol	.20	.....	.....	.....	.....
30. Ash	.1480	.1560	.144	.160	.160
31. Phosphoric acid, gravimetric	.0487	.044	.035	.068	.....
32. Phosphoric acid, uranium volumetric	.0487	.....	.....	.....	.035
33. Protein	0.31	0.26	0.27	0.304	0.33
34. Sulphurous acid (mg per liter)	.....	.....	.....	21.92	19.82
35. Fluorids	Present.	Present.	Present.	.....	Present.

TABLE 1.—*Analytical results obtained by cooperating chemists on referee's sample of beer No. 1*—Continued.

Determinations.	O. S. Marckworth.	W. B. Pope.	A. Lasché.	Norris Thompson.	H. E. Bishop.
1. Specific gravity at 15° C	1.0112	1.0108	1.0109	1.0108	1.0109
2. 20° C	1.0109	1.0108	1.0107	1.0116	1.0109
3. Alcohol A: by distillation (per cent by weight).	3.85	3.89	3.96	3.96	3.96
4. C: $A=B_1 \div B_2$ (per cent by weight)	3.68	.....	3.73	3.76	3.72
5. D: = by the immersion refractometer	.....	.....	3.97	4.03	4.06
6. E: by $A=(B_1-B_2)+15$	.....	.....	4.15	4.15	3.93
7. Extract A: by direct weighing	4.46	3.86	4.50	4.64	4.51
8. B: Sp.=g+(l—a)	4.74	.....	4.50	4.88	4.69
9. C: from dealcoholized beer	4.68	4.58	4.46	4.50	4.61
10. D: by immersion refractometer	.....	.....	4.42	4.41	4.12
11. Extract of wort A: O=2A+E	12.44	12.36	12.34	12.60	12.53
12. B: O=(A×1.92)+E	12.13	12.00	12.05	12.26	12.21
13. C: O=E+[(E−A)÷(1.232−1)]	.....	.....	.....	.....	.....
14. D: O=100[(2.0665×A)+E] ÷ [(1.0665×A)+100]	.....	.....	.....	.....	.....
15. E: G=sp+si	11.35	.....	11.57	.....	.....
16. Specific gravity, Shultz & Ostermann's tables	1.0493	.....	1.0489	1.0499	1.0483
17. Ellion's table	1.0523	.....	1.0520	1.0530	1.0513
18. Balling's table	1.0507	.....	1.0503	1.0514	1.0497
19. Degree of fermentation: (200 A)÷B	61.53	.....	63.85	66.00	63.22
20. Total acids A: gram lactic acid per 100 cc	.1162	.054	.058	.120	.099
21. B: cc $\frac{n}{10}$ NaOH to 100 cc	12.8	6.0	6.5	13.0	11.5
22. Volatile acids A: gram acetic acid per 100 cc	.0045	.0061	.003	.006	.0045
23. B: cc $\frac{n}{10}$ NaOH to 100 cc	.75	1.02	.5	1.0	.6
24. Reducing sugars	2.02	.....	1.418	1.544	1.693
25. Dextrin A: by Allihn's method	.....	.....	1.990	2.7	1.6
26. B: =(A−(M×8.1))÷(11.6)	1.77	.....	.....	2.5	1.57
27. Direct polarization: °V	32.8	31.8	.....	31.9	32.0
28. Invert polarization: °V	32.55	31.68	.....	31.6	32.8
29. Glycerol	.....	.....	.11	.....	.....
30. Ash	.145	.165	.160	.14	.155
31. Phosphoric acid, gravimetric	.....	.....	.....	.....	.....
32. Phosphoric acid, uranium volumetric	.....	.038	.047	.047	.042
33. Protein	.....	.....	.547	.244	.259
34. Sulphurous acid (mg per liter)	.....	.....	22.0	21.30	.....
35. Fluorids	.....	Present.	.....	Present.	Present.

TABLE 2.—*Analytical results obtained by cooperating chemists on referee's sample of beer No. 2.*

[Results calculated to grams per 100 cc unless otherwise stated.]

Determinations.	W. M. Cross.	Francis Wyatt.	Max Henius.	Frank Kremer.	B. Breitenfeld.
1. Specific gravity at 15° C	1.0123	1.0131	1.0129	1.0310	1.01265
2. 20° C	1.0114	1.0129	1.0127	1.0122	1.01257
3. Alcohol A: by distillation (per cent by weight).	4.45	3.81	3.89	3.62	3.91
4. C: $A=B_1 \div B_2$ (per cent by weight)	4.00	3.63	.....	3.80	3.83
5. D: by the immersion refractometer	4.63	3.93	3.83	.....	3.82
6. E: by $A=(B_1-B_2)+15$	4.03	.....	.....	.....	.....
7. Extract A: by direct weighing	5.483	5.104	4.86	5.426	4.98
8. B: Sp.=g+(l—a)	5.39	5.24	5.23	5.10	4.91
9. C: from dealcoholized beer	4.90	5.17	5.16	5.23	4.91
10. D: by immersion refractometer	5.00	.....	.....	.....	4.94
11. Extract of wort A: O=2A+E	14.48	12.95	12.94	12.43	12.73
12. B: O=(A×1.92)+E	14.12	12.70	12.628	12.04	12.41
13. C: O=E+[(E−A)÷(1.232−1)]	.....	13.31	13.04	12.50	.....
14. D: O=100[(2.0665×A)+E) ÷ [(1.0665×A)+100]	.....	12.78	12.10	.....	.....
15. E: G=sp+si	14.15	12.12	12.38	11.77	.....
16. Specific gravity, Shultz & Ostermann's tables	1.0532	1.0503	1.0500	1.0492	1.04685
17. Ellion's table	1.0564	1.0521	1.0476	1.0522	1.0495
18. Balling's table	1.0592	1.0505	1.0505	1.0506	1.05053
19. Degree of fermentation: (200 A)÷B	63.1	59.5	60.12	5.68	63.0
20. Total acids A: gram lactic acid per 100 cc	.166	.085	.045	.131	.120
21. B: cc $\frac{n}{10}$ NaOH to 100 cc	18.5	9.5	5.0	14.5	13.0
22. Volatile acids A: gram acetic acid per 100 cc	.0064	.009	.006	.003	.007
23. B: cc $\frac{n}{10}$ NaOH to 100 cc	1.07	1.5	1.0	.5	.75
24. Reducing sugars	1.57	1.39	1.36	1.27	1.40
25. Dextrin A: by Allihn's method	2.9	.....	.....	.....	2.30
26. B: =(A−(M×8.1))÷(11.6)	2.1	.....	.....	.....	2.20
27. Direct polarization: °V	32.2	.....	.....	.....	36.8
28. Invert polarization: °V	31.5	.....	.....	.....	26.4
29. Glycerol	.21	.....	.....	.....	.....
30. Ash	.130	.156	.158	.16	.16
31. Phosphoric acid, gravimetric	.0406	.042	.039	.068	.....
32. Phosphoric acid, uranium volumetric	.0496	.....	.....	.....	.035
33. Protein	0.31	.256	.266	.294	.36
34. Sulphurous acid (mg per liter)	.....	Absent.	Absent.	15.07	.....
35. Fluorids	.....	Absent.	Absent.	.....	.....

TABLE 2.—*Analytical results obtained by cooperating chemists on referee's sample of beer No. 2.*—Continued.

Determinations.	O. S. Marckworth.	W. B. Pope.	A. Lasché.	Norris Thompson.	H. E. Bishop.
1. Specific gravity at 15° C	1.01303	1.0127	1.0130	1.0126	1.0123
2. 20° C	1.01278	1.0128	1.427	1.0135	1.0122
3. Alcohol A: by distillation (per cent by weight)	3.78	3.86	3.88	3.96	3.60
4. C: $A=B_1 \div B_2$ (per cent by weight)	4.10	.....	3.64	4.09	3.70
5. D: by the immersion refractometer	.....	.....	3.90	3.98	3.78
6. E: by $A=(B_1-B_2)+15$	.....	.....	4.12	4.21	4.03
7. Extract A: by direct weighing	4.92	4.35	4.96	5.21	4.844
8. B: $Sp.=g+(1-a)$	5.20	4.93	5.00	4.92	4.88
9. C: from dealcoholized beer	5.36	5.09	4.95	5.16	4.93
10. D: by immersion refractometer	.....	.....	4.86	4.92	4.63
11. Extract of wort, A: $C=2A+E$	12.76	12.81	12.71	13.09	12.08
12. B: $C=(A\times 1.92)+E$	12.96	12.50	12.40	12.769	11.79
13. C: $C=E+[(E-A)\div(1.232-1)]$	.....	.....	.....	.....	.....
14. D: $C=100[(2.0665+A)+E)]\times[(1.0665\times A+100)]$	.....	.....	.....	.....	.....
15. E: $G=sp+si$	11.78	.....	11.95	.....	.....
16. Special gravity, Shultz and Ostermann's tables.	1.0506	.....	1.0503	1.0518	1.0478
17. Ellion's table	1.0537	.....	1.0535	1.0551	1.0507
18. Balling's table	.....	.....	1.0518	1.0534	1.0492
19. Degree of fermentation: $(200A)\div B$	58.47	.....	61.0	62.3	59.60
20. Total acids A: gm cc lactic acid per 100 cc	.1215	.054	.072	.120	.099
21. B: cc $\frac{n}{10}$ NaOH to 100 cc	13.5	6.0	8.0	13.0	11.0
22. Volatile acids A: gm acetic acid per 100 cc	.0045	.0107	.003	.006	.0045
23. B: cc $\frac{n}{10}$ NaOH to 100 cc	.75	1.78	.5	1.0	.6
24. Reducing sugars	1.80	.....	1.233	1.373	1.98
25. Dextrin A: by Allihn's method	.....	.....	.....	2.21	1.8
26. B: $=(A\div(M\times 8.1)-(11.6)$	2.41	.....	.....	2.27	1.75
27. Direct polarization: °V	38.94	38.3	.....	37.4	37.2
28. Invert polarization: °V	39.60	38.28	.....	38.3	38.7
29. Glycerol	.....	.....	.....	.....	.....
30. Ash	.14	.168	.166	.18	.145
31. Phosphoric acid: gravimetric	.....	.....	.....	.....	.....
32. Phosphoric acid: uranium volumetric	.....	.040	.0517	.045	.040
33. Protein	.....	.....	.498	.308	.275
34. Sulphurous acid (mg per liter)	.....	.....	.....	.....	Absent.
35. Fluorids	.....	.....	Present.	.....	Absent.

TABLE 3.—*Average results and maximum and minimum variations obtained by chemists on referee's samples of beer Nos. 1 and 2.*

Determinations.	Sample 1.			Sample 2.		
	Average.	Highest variation.	Lowest variation.	Average.	Highest variation.	Lowest variation
1. Specific gravity at 15° C	1.0109	0.0004	0.0004	1.0147	0.0004	0.0004
2. at 20° C	1.0108	.0004	.0002	1.0125	.0010	.0011
3. Alcohol method A	3.88	.08	.13	3.81	.15	.36
4. Alcohol method C	3.76	.16	.08	3.85	.25	.22
5. Alcohol method D	3.92	.14	.09	3.87	.11	.09
6. Alcohol method E	3.94	.21	.42	4.04	.12	.06
7. Extract method A	4.54	.16	.15	5.014	.491	.664
8. Extract method B	4.63	.11	.14	5.04	.34	.41
9. Extract method C	4.60	.12	.14	5.08	.28	.18
10. Extract method D	4.50	.25	.08	4.87	.13	.24
11. Extract of wort A	12.36	.24	.43	12.72	1.01	.64
12. Extract of wort B	12.05	.21	.42	12.48	.44	.32
13. Extract of wort C	12.31	.42	.53	12.95	.45	.36
14. Extract of wort D	12.02	.29	.69	12.01	.41	.27
15. Extract of wort E	11.62	.43	.27	12.00	.38	.12
16. Specific gravity, Schultz and Ostermann's table	1.0480	.0013	.0026	1.0500	.0032	.0032
17. Specific gravity, Ellion's table	1.0503	.0027	.0043	1.0523	.0041	.0047
18. Specific gravity, Balling's table	1.0496	.0013	.0011	1.0519	.0073	.0072
19. Degree of fermentation	63.32	2.68	1.79	60.88	2.41	3.31
20. Total acids A	.092	.032	.038	.1013	.065	.056
21. Total acids B	10.21	3.59	4.21	11.2	7.5	6.0
22. Volatile acids A	.0050	.0011	.0020	.0060	.0047	.0030
23. Volatile acids B	.737	.313	.237	.945	.835	.445
24. Reducing sugars	1.582	.438	.180	1.49	.49	.26
25. Dextrin A	2.14	.56	.54	2.30	.60	.50
26. Dextrin B	1.81	.69	.43	2.14	.27	.39
27. Direct polarization	32.1	.8	.2	37.72	1.22	.92
28. Invert polarization	32.04	.76	.36	38.25	1.35	1.85
29. Glycerol	.16	.04	.05	.21	.....	.....
30. Ash	.1533	.012	.013	.1563	.0237	.0263
31. Phosphoric acid gravity	.0489	.0191	.0139	.0474	.0206	.0084
32. Phosphoric acid volatile	.0429	.0019	.0079	.0435	.0136	.0085
33. Protein	.315	.015	.071	.3208	.1172	.0648
34. Sulphurous acid	21.26	.74	1.44	.....	.....	.....
35. Fluorids	.....	.....	.....	.....	.....	.....

COMMENTS BY ANALYSTS.

K. v. Bauer-Breitenfeld.

Preparation of the samples.—The two methods—shaking, and pouring from one beaker into the other—showed practically no difference in the results, the specific gravity being the same according to both methods. The amount of alcohol, after shaking the sample, was only 0.01 per cent higher than after pouring. Both methods, furthermore, secure a complete separation of the carbonic-acid gas.

Determination of specific gravity.—We made all three determinations of specific gravity at the temperatures of 15°, 17.5°, and 20° C., using, of course, in the calculation three different water weights of the pycnometer, corresponding to the temperature employed. We did not find any important differences between the determinations, the specific gravity increasing only slightly with the lowering of the temperatures. The differences in the extreme cases amounted to—

0.00007 in the specific gravity of alcohol.
0.00011 in the specific gravity of the real extract.
0.00014 in the specific gravity of the beer itself.

As the difference between the two temperatures was never higher than indicated above, they can practically be substituted for one another. For this reason we would be in favor of the employment of the temperature of 20° C. as a normal temperature for the determination of specific gravities.

The tables of Ellion and Balling give almost the same results with respect to the amount of extract, while the table of Schultz and Ostermann indicates a somewhat higher extract.

Determination of alcohol.—We determined the alcohol by the distillation method strictly according to your suggestions, but having no Zeiss immersion refractometer at our disposal used instead of this the refractometer of Tornoe. The results obtained by this instrument, and by the distillation method, with regard to the amount of alcohol or real extract, never differed more than 0.1 per cent. Between the alcohol tables of Squibb and Baumhauer no considerable difference appears to exist.

Determination of extract.—No considerable difference between the methods of evaporating to dryness, distillation, and Tornoe's refractometer could be detected. The first method, however, takes too long a time to come into practical use.

Total acids.—Instead of using neutral litmus paper as an indicator, we prefer the red phenolphthalein solution, prepared by adding 12 drops of the ordinary alcoholic solution of phenolphthalein and 0.2 cc of normal alkali to 20 cc of water. The end point of the titration is determined by transferring one drop of the solution to a porcelain plate and adding one drop of the beer as soon as the red color of the indicator does not appear.

In answer to an inquiry, K. v. Baur-Breitenfeld submitted the following method for the determination of dextrin by means of the polariscope, together with other notes:

Twenty-five cubic centimeters of beer are clarified with 5 cc of alumina cream and filtered after the addition of water until the volume amounts to 50 cc. The clarified filtrate is polarized in the 200 mm tube, using the Soleil Ventzke apparatus. The reading in degrees is multiplied by 2 on account of the dilution, and from the total rotation is deducted the rotation of the corresponding amount of maltose in 100 cc of beer, determined by the gravimetric-analytical method. One gram of maltose dissolved in 100 cc in the 200 mm tube rotates 8.1+, and the quantity of maltose found must, therefore, be multiplied by 8.1.

After the deduction of this rotation from the total rotation, the result corresponds to the quantity of dextrin in 100 cc of beer. As 1 gram of dextrin in 100 cc of solution in the 200 mm tube rotates 11.6+, the quantity of dextrin is found by dividing the polarization-difference by 11.6.

Inasmuch as in fermented normal beer the reducing sugar consists almost exclusively of maltose and very small quantities of reducing malto-dextrins, it is possible to deduce from an abnormally low amount of dextrin obtained by the polarization method, that in the production of the beer, aside from malt and unmalted cereals, large quantities of dextrose (glucose and grape sugar) have been used.

As far as the use of the refractometer of Tornoe is concerned in connection with the examination of beer, we have found that the results obtained in this way correspond very well with those obtained by the analytical method. In our laboratory almost all the beers are examined according to both methods, which gives us an excellent control of our work and its accuracy.

Fluorids.—We have found that when no fluorid has been added to a beer, 200 cc of such beer, when tested for fluorin according to adopted method, will not show any appreciable etching on glass. We also found that barley in its natural condition did not contain enough fluorin in 10 grams to etch glass. If, therefore, fluorin is present in a beer to such an extent in 200 cc, that it can be made to etch glass, it is, in our opinion, safe to say that a fluorid has been added to the beer. When used, the fluorid is added in the proportion of one pound to 150 barrels, or approximately 0.0025 per cent. Of this fluorid two-thirds or 0.0016 per cent is fluorin, that is, 16 mg of fluorin per liter.

The coating on the glass is made of a mixture of paraffin and wax. The tested flat glass lies tight on top of the platinum crucible and a small tin vessel filled with chopped ice is placed on top of this glass. The markings in the wax are not to be done with a lead pencil, but with a pointed piece of soft wood.

A slight etching not visible in the reflected light, but visible when breathing upon the glass, should not be considered a fluorin reaction, as such reaction, in some instances, was obtained when absolutely pure sulphuric acid was employed, and even a slight etching visible in the reflected light without breathing upon the glass does not necessarily indicate the addition of fluorids, since these have been found to be a normal constituent of many waters and brewing materials.

Sulphites.—According to Graf,[a] analyses made by him showed in ordinary German and Austrian beers from 3 to 23 mg of sulphurous acid per liter; in Frankish beers from 0 to 89 mg per liter. As sulphured hops may introduce from 3 to 7 mg of sulphurous acid per liter and by action of yeast (according to Graf) appreciably more, say, 10 mg per liter, it is seen that 17 mg of sulphurous acid may be found in beer to which no sulphites have been added.

We have, in our laboratory, found 13 mg of sulphur dioxid in beers free from sulphites. As the amount of meta-bisulphite of potassium when used in beer is one pound to 60 barrels, or about 34 mg of sulphur dioxid per liter, it is safe to say that when only one half as much, or 17 mg per liter, is found, no sulphites have been added. Since this quantity will not preserve, and the addition of a corresponding amount of sulphites would therefore be barren of result, any amount up to this limit should be allowable.

COMMENTS BY ASSOCIATE REFEREE.

The results as compiled show two things clearly. They express the value of standard methods for analytical work. The ten analysts working upon a sample of beer which, although pasteurized, was still subject to change in composition during the weeks or months that lapsed before the analysis was made, obtained for the most part very concordant results, the personal error usually being so small as to be negligible. On the other hand, the determination of the several factors by the different methods showed marked variations. In the preparation of the sample, the same result was obtained when the carbon dioxid was removed by shaking, as when the beer was poured from one beaker to another. Either method removes the carbon dioxid completely. The specific gravity of the beer is practically the same at 15° C. as at 20° C. when compared with water at the same temperature, there being but 0.0001 difference in the average results.

The various methods suggested for the determination of alcohol give fairly concordant results. The use of the Zeiss immersion refractometer gives results practically identical with those obtained by taking the specific gravity of the distillate. The only precaution necessary is that of watching the temperature at the time of reading.

When it is not possible to distil the alcohol a correct estimation may be made by use of the formula $A=(B_1-B_2)+15$, when $A=$ reading on immersion refractometer, $B_1=$ reading of beer, $B_2=$ reading

[a] Zts. für das gesammte Brauwesen, 1904, *27*: 617.

of dealcoholized beer, the value for A being determined from alcohol tables accompanying the instrument.

The extract determinations gave fairly concordant results. The use of the immersion refractometer for this determination also gives accurate results. The dehydration of maltose in the estimation of extract by direct weighing is apparently more a theoretical than an actual error. The determinations of extract and specific gravity of wort by different analysts agree very well, the greatest error being that introduced by the use of different tables. The formulas B, C, D, and E offer no advantage over the more simple formula $O=2A+E$. This formula is considered by brewers to express very nearly what takes place in practice.

Kremer[a] has made a careful study of the different formulas, employing them in eight experimental brews of different character. He states that although the formula $O=2A+E$ is not correct from a scientific standpoint, since theoretically two parts of maltose are not required to produce one part of alcohol, yet in practice it is the most accurate. The specific gravity of the wort varies greatly according to the table employed. Schultz and Ostermann's and Balling's tables agree closely, Ellion's tables giving somewhat higher results.

The degree of fermentation calculated according to the formula $D=\frac{200A}{B}$ gives entirely satisfactory results. The determination of total acids is not entirely satisfactory, since the end point with neutral litmus paper is obscure. The use of phenolphthalein is suggested as a preferable indicator. As the end point is reached a drop of the beer is added to a drop of phenolphthalein on a porcelain plate; in this way a sharp end point is readily obtainable.

The volatile acid, as determined, did not give concordant results. The error in the case of both total and volatile acids may be due to the age of the sample when analysis was made.

No comment is necessary in regard to reducing sugars, as the results are entirely satisfactory.

The same is true of the dextrin determinations. Good results are obtained by the use of the formula $D=\frac{A-(M\times 8.1)}{11.6}$ when D= dextrin in grams per 100 cc; A, total rotation in degrees Ventzke in 200 mm tube, and M, percentage of maltose as determined gravimetrically. This formula is based upon the difference of optical activity of dextrin and maltose, and its use furnishes a ready and accurate method for determining a factor which is otherwise somewhat difficult to obtain.

It is impossible to explain the differences in polarization obtained by the various analysts, except on the assumption that the various instruments were not in accord.

The results of ash estimation are very close.

The phosphoric acid results obtained, both volumetrically and gravimetrically, agree very well. The volumetric method, with standard uranium solution, is to be preferred on account of the rapidity with which it may be used.

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[a] Letters on Brewing, vol. 1, p. 9; vol. 1, No. 2, p. 19.

All the analysts reporting on fluorids had no difficulty in detecting its presence in sample No. 1.

The sulphurous acid estimation in the same sample also gave satisfactory results.

CONCLUSIONS AND RECOMMENDATIONS.

It is greatly to be regretted that there are in use several tables for the determination of alcohol and extract, and that the estimations are not made by different analysts at a standard temperature. The investigations undertaken have not been arranged so as to show which of the various tables is the most reliable, nor what temperature is the most suitable for alcohol estimations. The results, however, do indicate the necessity of the adoption of standard tables and a standard temperature. The brewing schools all employ the Balling extract table; Bulletin 65 gives both Schultz and Ostermann's and Ellion's tables. If one of the tables is to be preferred above the other it should be recognized as the standard table. In 1903 President Davidson, of the association of official agricultural chemists, in his annual address recommended that the temperature at which apparatus is graduated should be changed to one more nearly in accordance with room temperature. In 1904 the referee on beer repeated the recommendation and called attention to a revision of the alcoholometric tables. The use of a standard temperature of 20° C. seems to be growing rapidly in this country. It is being employed by the Bureau of Standards and many food chemists, and it can be but a short time before it will be the accepted standard temperature. The referee therefore suggests that a committee be appointed by this association for the purpose of revising the alcoholometric tables now given in Bulletin 65, that they may be adopted for use at this new temperature.

It is further recommended that the following methods of beer analysis be adopted as official for this association.

PROPOSED OFFICIAL METHODS OF BEER ANALYSIS.

1. Preparation of Sample.

Transfer the contents of the bottle or bottles to a large flask and shake vigorously to hasten the escape of carbon dioxid, care being taken that the beer is not below 15° C., since below this temperature the carbon dioxid is retained in the beer and is liable to form bubbles in the pycnometer.

2. Specific Gravity.

Determine specific gravity at 20° C. by means of the pycnometer, by a small accurately graduated hydrometer, by a Westphal balance, or by a Westphal plummet on the analytical balance.

3. Alcohol

(a) distillation method.

Measure 100 cc of the liquid at 20° C. into a round-bottomed distillation flask, add 50 cc of distilled water, and if the beer is markedly acid, 0.1 or 0.2 gram of precipitated calcium carbonate, connect with condenser, and distil off 90 cc into an accurately graduated 100 cc stoppered flask, care being taken to prevent loss of alcohol during the distillation. This is best accomplished by inserting a two-holed rubber stopper in the mouth of the flask, through one hole of which passes an adapter which is tightly filled to the end of the condenser and through the other a small funnel filled

with glass beads kept moist by sprinkling with water. Wash down the beads several times during the distillation with a small quantity of water, thus saving any alcohol that may have collected on the moist surface. Bring the contents of the receiver to 20° C. make up to the mark with distilled water, determine the specific gravity as directed under specific gravity and obtain the corresponding percentage of alcohol by volume and grams per 100 cc, from standard tables at 20° C. Calculate results to per cent by weight by dividing the results expressed as grams per 100 cc by the specific gravity of the original sample.

(B) OPTIONAL METHOD

Calculate the alcohol content from the reading of the Zeiss immersion refractometer on the distillate, reporting the results at 20° C.

4. EXTRACT.

(A) Evaporate 25 cc of the beer in a tared platinum dish to constant weight in a water oven at 80° C.

(B) OPTIONAL METHOD NO. 1.

Calculate according to formula $sp = g + (1 - a)$, in which sp is the specific gravity of the beer, a the specific gravity of the distillate obtained in the determination of alcohol, and determine value of sp from standard extract of beer-wort tables.

(C) OPTIONAL METHOD NO. 2.

Take immersion refractometer reading of dealcoholized beer and calculate extract in grams per 100 cc.

5. EXTRACT AND SPECIFIC GRAVITY OF ORIGINAL WORT.

Calculate percentage of extract from the formula $O = 2A + E$, when O = original extract of wort, A = alcohol by weight, and E = extract of dealcoholized beer. From extract calculated as above compute from standard tables the specific gravity of the wort.

6. DEGREE OF FERMENTATION.

Calculate from the formula $D = \frac{200A}{B}$ in which D is the degree of fermentation A = percentage of alcohol by weight, and B the original extract.

7. TOTAL ACIDS.

(A) Heat 20 cc of the sample to incipient boiling to liberate carbon dioxid, and, titrate with decinormal sodium hydroxid, using neutral litmus as indicator. Each cubic centimeter of decinormal alkali employed is equivalent to 0.009 grams of lactic acid. The number of cubic centimeters of decinormal alkali employed in titrating 20 cc of beer is multiplied by 0.045 for the acidity expressed as grams of lactic acid per 100 cc.

(B) Calculate the cubic centimeters of decinormal sodium hydroxid required to neutralize the acidity of 100 cc of the sample.

8. VOLATILE ACIDS.

(A) The volatile acid, as acetic acid, is determined by titrating 20 cc of the alcoholic distillate with decinormal sodium hydroxid solution, using phenolphthalein as an indicator. The number of cubic centimeters of decinormal alkali employed multiplied by 0.030 gives the acidity expressed as grams of acetic acid per 100 cc.

(B) Calculate the cubic centimeters of decinormal sodium hydroxid required to neutralize the acidity of 100 cc of sample.

9. REDUCING SUGARS.

Twenty-five cc of beer, free from carbon dioxid, is diluted with water to 100 cc. The reducing sugar is determined in 25 cc of this solution, as directed on page 49 of Bulletin 65, the solution being boiled four minutes instead of two. Express the results in terms of maltose equivalent to copper reduced, according to Table IX, page 144, Bulletin 65.

10. Dextrin.

(a) sachsse-allihn method.

Employ Sachsse's method for the hydrolization of starch and determine dextrose according to Allihn, as follows:

Fifty cc of beer and 15 cc of hydrochloric acid, specific gravity 1.125, are diluted to 200 cc, attached to a reflux condenser and kept in a boiling water bath for two hours. Neutralize with caustic soda and dilute to 250 cc, or 300 cc in a beer with high extract. Add 25 cc of the solution to the boiling Fehling solution and boil two minutes. Multiply the oxid of copper found by 0.8 to obtain the corresponding amount of copper and refer to Allihn's dextrose table (Table VIII, p. 143, Bul. 65). The amount of dextrose thus found multiplied by 20 (or 24 if diluted to 300 cc) and divided by the specific gravity equals the dextrose in the original beer. From this figure subtract 95 per cent of the amount of maltose in the original beer and multiply the remainder by 0.9, the result being the percentage of dextrin in the original wort.

(b) optional method.

Dextrin may be determined by the following method, based on the difference between its optical activity and that of maltose.

$D=\frac{A-(M\times 8.1)}{11.6}$ when D=dextrin, in grams per 100 cc, A=total rotation in degrees Ventzke in 200 mm tube, and M=percentage of maltose as determined gravimetrically.

11. Direct Polarization.

Read the polarization of the original sample in degrees Ventzke in a 200 mm tube. If the beer is turbid, clarify by shaking with alumina cream.

12. Invert Polarization.

To 10 cc of the beer in a small flask add 1 cc of concentrated hydrochloric acid, invert by slowly heating to 68° C. cool, polarize in a 200 mm tube, and increase the reading one-tenth to allow for dilution.

13. Glycerol.

Proceed as directed on page 62, Bulletin 65. The milk of lime is added during evaporation after the carbon dioxid has been expelled. It is advisable that the filtrate after being evaporated to a sirupy consistency be treated again with 5 cc of absolute alcohol and 2 portions of 7.5 cc each of absolute ether. If clear, continue as directed. If not clear, it is necessary to treat again as below.

14. Ash.

Evaporate 25 cc of the sample to dryness, ignite at low redness until thoroughly charred, place in a muffle and continue the ignition until ash is white. Leaching is rarely necessary.

15. Phosphoric Acid.

Measure out 50 cc of the original beer, free from carbonic acid, into a small beaker. Add 5 cc of an acid solution of sodium acetate and heat to boiling. Run in with a burette, standard uranium solution, 0.5 cc at a time, testing each time until a drop of the beer when placed on a white plate colors a drop of potassium ferrocyanid slightly brown. The number of cubic centimeters of the uranium-acetate solution necessary, multiplied by 0.01 gives the grams per 100 cc of phosphoric acid in the beer. If the beer is very dark, employ the official gravimetric or volumetric method, using the residue obtained in the determination of the ash.

16. Protein.

Evaporate 25 cc of the original beer, to which has been added a small amount of tannin to prevent frothing, and proceed according to Kjeldahl or Gunning method for the determination of nitrogen and multiply the result by 6.25.

17. Carbon Dioxid.

See Bulletin 65, p. 95, 1902.

18. Preservatives.

Add 5 cc of dilute sulphuric acid to 100 cc of beer from which the alcohol has been driven off, and shake out with an equal volume of a mixture of ether and benzene. Pour off the supernatent liquid and test for salicylic acid, benzoic acid, and saccharin in the usual manner. It is well to reserve 2 or 3 cc of the liquid in a test tube for the salicylic-acid test before exaporating the remainder to dryness in a small porcelain evaporating dish.

(a) sulphites and sulphurous acids.

Place 25 cc of a solution of potassium hydroxid containing 56 grams per liter in a flask of approximately 200 cc capacity. Introduce 50 cc of beer by means of a pipette, mix with the potassium hydroxid, and allow the mixture to stand for fifteen minutes with occasional agitation. Add 10 cc of 1-3 sulphuric acid and a few cubic centimeters of starch solution, and titrate the mixture with a fiftieth-normal iodin solution. Introduce the iodin solution as rapidly as possible and continue the addition until the blue color will last several minutes. One cubic centimeter of fiftieth-normal iodin solution is equivalent to 0.00064 gram of sulphur dioxid. The number of cubic centimeters of the iodin solution employed, multiplied by 0.00128, gives the weight of the total sulphur dioxid expressed in grams per 100 cc.

(b) fluorids (method of blarez).

Thoroughly mix the sample and heat 150 cc to boiling. Add to the boiling liquid 5 cc of a 10 per cent solution of of barium acetate. Collect the precipitate in a compact mass, using to advantage a centrifuge, wash upon a small filter and dry in the oven. Transfer to a platinum crucible, first breaking up the dry precipitate and then adding the filter ash to crucible. Prepare a glass plate (preferably of the thin variety commonly used for lantern-slide covers) as follows: First thoroughly clean and polish and coat on one side by carefully dipping while hot in a mixture of equal parts of Carnaüba wax and paraffin. Near the middle of the plate make a small cross or other distinctive mark through the wax with a sharp instrument, such as a pointed piece of wood or ivory, which will remove the wax and expose the glass without scratching the latter. Add a few drops of concentrated sulphuric acid to the residue in the crucible and cover with the waxed plate, having the mark nearly over the center and making sure that the crucible is firmly imbedded in the wax. Place in close contact with the top or unwaxed surface of the plate a cooling device consisting of a glass cylinder the bottom of which is a thin sheet of pure rubber. Keep the cylinder filled with ice water so that the wax does not melt. Heat the bottom of the crucible gently over a low flame or on an electric stove for an hour. Remove the glass plate and indicate the location of the distinguishing mark on the unwaxed surface of the plate by means of gummed strips of paper, melt off the wax by heat or a jet of steam and thoroughly clean the glass with a soft cloth. A distinct etching will be apparent on the glass where it was exposed if a fluorid be present.

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